(12) UK Patent Application (19) GB (11) 2 190 921 (13) A

(43) Application published 2 Dec 1987

(21) Application No 8612838

(22) Date of filing 27 May 1986

(71) Applicant
Unilever PLC,

(Incorporated in United Kingdom),

Unilever House, Blackfriars, London EC4P 4BQ

(72) Inventors
Graham Andrew Catton,
Paul Michael Hill

(74) Agent and/or Address for Service
D. Gambell, Patent Division, Unilever PLC, PO Box 68,
London EC4

(51) INT CL[4]
C11D 11/0

(52) Domestic classification (Edition I)
C5D 6A5B 6A5C 6A5D1 6A5D2 6A5E 6A8B 6A9
6B12G2A 6B12N1 6B8 6C6 6D

(56) Documents cited
None

(58) Field of search
C5D
Selected US specifications from IPC sub-class C11D

(54) Granular detergent composition

(57) A process for preparing a granular detergent composition, especially for fabric washing, comprises mixing a first granular component containing more than 8% nonionic detergent and more than 20% inorganic salt with a second granular component which is particles of a fabric softening clay agglomerated with water. Both components have a particle size of 150-2000 microns. The clay particles are less than 150 microns in size. The first component may be made by spray drying and the inorganic salt may be a detergency builder. The clay is preferably a smectite. The process reduces the negative effect of the detergent on the performance of the clay.

GB 2 190 921 A

SPECIFICATION

Process for preparing a detergent composition

This invention relates to a process for preparing a detergent composition, in particular a detergent composition for washing fabrics and providing said fabrics with a softening benefit.

It is common practice to wash fabrics in detergent compositions which contain a detergent active material for removing soil from the fabrics. With some fabrics, especially of natural origin, repeated washing can lead to fabric harshness, giving the fabric an unpleasant feel. For some years fabric conditioning products have been available, intended *inter alia* for alleviating this fabric harshness by softening the fabrics in a post washing step, eg in the rinse step of a fabric laundering process. There has been a desire to provide a single detergent composition which would be capable of both washing and softening fabrics to overcome the inconvenience of using separate products.

According to GB 1 400 898 (Procter & Gamble) a possible solution to this problem is to include in the detergent composition a three-layer smectite clay containing material having a cation exchange capacity of at least 50 meq/100g together with an anionic or similar detergent active material.

According to GB 1 462 484 (Procter & Gamble), such clays are almost ineffective in the presence of significant amounts of nonionic detergents, and that such clays should therefore be replaced by modified clays which carry a proportion of quaterniary ammonium cations. Such modified clays are generally more costly than their un-modified counterparts and have not been widely used.

However, detergent compositions containing significant amounts of nonionic detergent active material can be beneficial. Thus, for example, such compositions generally generate less foam in use. They are therefore more suitable for use in automatic washing machines and the need to include foam control agents in such compositions is reduced or removed. Further, compositions containing high levels of nonionic detergent active material are especially effective on removing greasy or oily soils from fabrics.

It is therefore an object of the present invention to provide a process for preparing detergent compositions containing high levels of nonionic detergent active material and clay as a fabric softener, while reducing or overcoming the negative effects of the nonionic active on the performance of the clay. We have now surprisingly discovered that this object can be achieved by preparing the composition in such a manner as to keep the nonionic detergent active material and the clay separate from each other. While one might suppose that in a wash liquor formed by adding such a composition to water, the nonionic detergent active material and the clay could interact, we have found that this does not occur, at least not to a significant extent within the time period of the washing process.

Thus according to the invention there is provided a process for preparing a granular detergent composition comprising the steps of:

(i) preparing a first granular component having a particle size of from 150 to 2000 microns comprising more than 8% by weight of a nonionic detergent active material and more than 20% by weight of an inorganic salt, said percentages being based on the weight of the final composition;

(ii) preparing a second granular component by agglomerating a fabric softening clay containing material having a particle size of less than 150 microns with water to form agglomerates having a particle size of from 150 to 2000 microns;

(iii) mixing said first and second granular components in the substantial absence of water.

The first granular component may be made by granulation but a process involving spray drying is preferred.

A preferred particle size is 200 to 1000 microns. The term "particle size" as used herein means the weight average particle size as measured by sieve analysis and preferably the majority of particles have a size falling within the given range.

The level of nonionic detergent active in the first granular component is preferably less than 15%, such as from 8.5 to 12% by weight, based on the final composition.

Suitable nonionic detergent compounds which may be used include in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide.

Specific nonionic detergent compounds are alkyl (C_6-C_{22}) phenols-ethylene oxide condensates, generally up to 25 EO, ie 25 units of ethylene oxide per molecule, the condensation products of aliphatic (C_8-C_{18}) primary or secondary linear or branched alcohols with ethylene oxide, generally up to 40 EO, and products may by condensation of ethylene oxide with the reaction products of propylen oxide and ethylendiamine. Other so-called nonionic detergent compounds include long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulphoxides.

The inorganic salt which is required to be present in the first granular component will normally be constituted by a detergency builder material, but other salts more usually known in the role of buffering agents (such as sodium silicate) or fillers (such as sodium sulphate) may also be used.

The detergency builder, when present, may be any material capable of reducing the level of free clacium ions in the wash liquor and will preferably provide the composition with other beneficial properties such as the generation of an alkaline pH, the suspension of soil removed from the fabric and the dispersion of the

fabric softening clay material. The level of the detergency builder may be from 20% to 70% by weight, most preferably from 25% to 50% by weight.

Examples of detergency builders include precipitating builders such as the alkali metal carbonates, bicarbonates, ortho phosphates, sequestering builders such as the alkali metal tripolyphosphates or ion-exchange builders such as the amorphous alkalimetal aluminosilicates or the zeolites. Organic detergency builders, such as alkali metal citrates or nitrilotriacetates may be present in the first granular component.

The clay containing material may be any such material capable of providing a fabric softening benefit. Usually these materials will be of natural origin containing a three-layer swellable smectite clay which is ideally of the calcium and/or sodium montmorillonite type. It is preferable to exchange the natural calcium clays to the sodium form by using sodium carbonate, either before or during granulation, as described in GB 2 138 037 (Colgate). The effectiveness of a clay containing material as a fabric softener will depend *interalia* on the level of smectite clay. Impurities such as calcite, feldspar and silica will often be present. Relatively impure clays can be used provided that such impurities are tolerable in the composition.

The second granular component is prepared by agglomerating a clay containing material with water. The naturally occurring clay containing material is first milled to a particle size of less than 150 microns, preferably from 30 to 100 microns. At this stage any non-clay gritty material in the clay containing material may be removed. The fine particles are then agglomerated to the required size with water. Pure water is suitable, there being no necessity to add further binding agents. However, the presence of up to about 1% inorganic salts, including those derived from water hardness, in the water may be tolerated.

The level of the fabric softening clay material in the composition should be sufficient to provide a softening benefit, such as from 1.5% to 35% by weight, most preferably from 4% to 15% by weight, calculated on the basis of the clay material *per se*, and therefore the level of agglomerated clay containing material should be chosen accordingly.

The particle sizes of the first and second granular components should preferably be approximately the same so as to prevent undue segregation after mixing. Thus it is preferred that the ratio of the particle sizes of the two components is within 2:1 to 1:2, most preferably within 1.5:1 to 1:1.5.

The two granular components should be mixed in the substantial absence of water. Thus the first granular component should not have a free-water content which is so high as to cause the particles thereof to be sticky, i.e. when mixing occurs the first granular component should be in the form of a free flowing powder. The clay granules preferably have a moisture content of less than 15%, preferably less than 12%, where the term "moisture" is that water which is lost on drying the clay granules to 135°C. In particular it is most desirable not to add any free water during the mixing step. The mixing may be carried out at room temperature or at a temperature not significantly higher than the melting point of the nonionic detergent active material, such as less than about 50°C.

Apart from the ingredients already mentioned a number of optional ingredients may also be present, either in the first component or added separately to the compositions.

In particular other detergent active materials may be present. Despite the advantages of the nonionic detergent active, some additional benefits may be derived from the presence of low levels, ie up to about 1.5% by weight, of an anionic detergent active. The preferred anionic detergent compounds are sodium (C_{11}-C_{15}) alkyl benzene sulphonates and sodium (C_{16}-C_{18}) alkyl sulphates.

Examples of other additives which may be present in the composition include the lather boosters such as alkanolamides, particularly the monoethanolamides derived from palm kernel fatty acids and coconut fatty acids, lather depressants, oxygen-releasing bleaching agents such as sodium perborate and sodium percarbonate, peracid bleach precursors, chlorine-releasing bleaching agents such as tricloroisocyanuric acid, inorganic salts such as sodium sulphate, and, usually present in very minor amounts, flourescent agents, perfumes, enzymes such as proteases and amylases, germicides and colourants.

The invention will now be illustrated by the following non-limiting examples:

Example 1

A first granule was prepared according to the following formulation:

Ingredient	Parts by weight
Anionic detergent active	1.5
Nonionic detergent active	8.5
Alkaline sodium silicate	7.5
Sodium tripolyphosphate	20.0
Sodium sulphate	10.0
Minor ingredients	2.2

The nonionic active used was Dobanol 25-7EO (Trade Mark) (ex Shell). The anionic active was sulphonated Dobane 113 (ex Shell). This granule was prepared by spray drying a slurry containing the listed ingredients, to an average particle size of 500 microns and a total water content of 7.5 parts.

To these granules were added 10 parts of agglomerated bentonite having an average particle size of 700 microns and a moisture content of 1.2 parts (12%). The clay used was sodium bentonite ex Steetley. Also

added at this stage were 16 parts of sodium perborate tetrahydrate, 9 parts of sodium carbonate, 3.75 parts of neutral sodium orthophosphate, about 3.75 parts of sodium sulphate and 0.3 parts enzymes making a total product of 100 parts by weight.

Terry towelling cotton test cloths were then washed in an automatic washing machine using a dosage of 120g product (the machine capacity was 20 1). The water hardness was 26° FH (equivalent to 26×10^{-4} molar free calcium ions). The washing temperature was 95°C and the washing time was 55 minutes. After washing and rinsing, the fabrics were line dried at 60% RH constant humidity and 21°C and assessed for softness.

The experiment was repeated with various modifications as follows:

(a) the clay was processed in the slurry together with other components of the first granule

(b) the clay was ommitted

(c) the clay was omitted and the ratio of anionic to nonionic active was increased to 6:4.

In all cases the softness of the fabrics was found to be significantly better with the composition of Example 1 compared with any of alternatives (a), (b) or (c).

When these experiments were repeated at 40°C using a wash time of 20 minutes, similar conclusions could be drawn from the results.

CLAIMS

1. A process for preparing a granular detergent comprising the steps of:

(i) preparing a first granular component having a particle size of from 150 to 2000 microns comprising more than 8% by weight of a nonionic detergent active material and more than 20% by weight of an inorganic salt, said percentages being based on the weight of the final composition;

(ii) preparing a second granular component by agglomerating a fabric softening clay containing material having a particle size of less than 150 microns with water to form agglomerates having a particle size of from 150 to 2000 microns;

(iii) mixing said first and second granular components in the substantial absence of water.

Printed for Her Majesty's Stationery Office by Croydon Printing Company (UK) Ltd, 10/87, D8991685.
Published by The Patent Office, 25 Southampton Buildings, London WC2A 1AY, from which copies may be obtained.